

Appl. No. 09/910,968
Appellant's Brief dated July 1, 2005
Notice of Appeal filed May 13, 2005



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**PATENT
IN THE UNITED STATES PATENT AND TRADEMARK OFFICE
BEFORE THE BOARD OF PATENT APPEALS AND INTERFERENCES**

Appl. No. : 09/910,968
Applicant : Brigitte BENAGE et al.
Filed : July 23, 2001
Title : RECYCLE OF NITROXYL-CONTAINING STREAMS AT LOW
TEMPERATURE
TC/A.U. : 1764
Examiner: : Nguyen, T.M.
Docket No. : 0036-PA (UNI057US)

APPELLANT'S BRIEF

Sir:

The above-identified Appellants submit this Appellant's Brief in triplicate pursuant to 37 C.F.R. § 1.192. The Notice of Appeal was filed on May 13, 2005.

Please charge the official fee of \$500.00 for filing a brief in support of an appeal to our Deposit Account Number 23-2656. A duplicate copy of this page is enclosed.

The Appellant relies upon the following authorities and arguments to maintain the appeal.

1. Real Party In Interest

The real party in interest for this matter is the Appellant's assignee. The assignee and real party in interest is Crompton Corporation; Benson Road; Middlebury, CT 06749.

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By amendment under 37 C.F.R. § 1.116 filed April 28, 2003, Appellant attempted to amend claims 4, 16, 19, and 31. In an Advisory Action dated June 2, 2003, the Examiner indicated that the proposed amendments would not be entered on the ground that they raised new issues that would require further consideration and/or search.

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The present invention is directed to an improvement in a process for the production and purification of unsaturated monomers employing distillation means and a nitroxyl-containing polymerization inhibitor of said monomers, wherein a process stream containing the nitroxyl-containing inhibitor is removed downstream of the distillation means and returned to the process ahead of the distillation means, wherein the improvement comprises recycling said stream containing the nitroxyl-containing inhibitor into the distillation means, wherein the temperature in the distillation means is no higher than about 110° C and the pressure is less than 760 mm Hg.

See pending claim 1, the specification at page 14, lines 1-5, and claim 8 of the application as originally filed.

6. Issue

Are claims 1, 2, 9, and 17-26 unpatentable over Arhancet (U.S. Patent No. 5,907,071) in view of Higgins et al. (U.S. Patent No. 4,033,829) under 35 U.S.C. 103(a)?

7. **Grouping of Claims**

All claims 1, 2, 9, and 17-26 are of a single group that is directed to an improvement in a process for the production and purification of unsaturated monomers employing distillation means and a nitroxyl-containing polymerization inhibitor of said monomers, wherein a process stream containing the nitroxyl-containing inhibitor is removed downstream of the distillation means and returned to the process ahead of the distillation means, wherein the improvement comprises recycling said stream containing the nitroxyl-containing inhibitor into the distillation means, wherein the temperature in the distillation means is no higher than about 110° C and the pressure is less than 760 mm Hg.

All the claims of the rejected group of claims stand or fall together.

8. **Argument**

Issue: Are claims 1, 2, 9, and 17-26 unpatentable over Arhancet (U.S. Patent No. 5,907,071) in view of Higgins et al. (U.S. Patent No. 4,033,829) under 35 U.S.C. 103(a)?

Arhancet discloses the inhibition of the polymerization of vinyl aromatic monomers such as styrene by the addition of a composition of a stable hindered nitroxyl radical and an oxime compound.

The Examiner has stated during prosecution:

"Arhancet discloses a method for inhibiting premature polymerization of vinyl aromatic monomers. The monomers are distilled in the presence of nitroxyl inhibitors (2,2,6,6-tetramethyl-1-piperidinoxy) at a temperature of 110° C and under

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Regarding claim 1, Arhancet does not disclose a step of recycling the inhibitor to the distillation column."

This last sentence is precisely the point. Arhancet teaches only using fresh nitroxyl compounds (i.e., not recycled) in combination with an oxime compound. As pointed out on page 13 of the present application, at lines 20-23,

"It is known in the industry that the recycling of streams utilizing nitroxyls as polymerization inhibitors in plants employing temperatures in excess of about 115°C causes loss of inhibitor efficiency, such that the tar recycle leads to a higher polymer content than would be expected or desirable."

The present invention is directed to the discovery of a means to substantially overcome this problem of the decreased efficiency of recycled *nitroxyl* inhibitors.

In an attempt to overcome this deficiency of Arhancet as a reference, the Examiner has cited the Higgins et al. reference.

According to the Examiner:

"Higgins discloses a process for production/purification of an unsaturated monomer feedstock by contacting the feedstock with inhibitors in a distillation/separation zone to produce a product stream containing the inhibitor and the unsaturated monomer which is then recycled back to the distillation/separation zone. Higgins also discloses that the process is operated on either a continuous or batch basis at an overhead pressure of the distillation

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column of 414 mm Hg and the product stream contains by-product impurities such as polymers. (See abstract; col. 1, lines 46-64; col. 3, line 11 through col. 6, line 66)

Regarding claim 1, Arhancet does not disclose a step of recycling the inhibitor to the distillation column. However, Higgins discloses a process for production/purification of an unsaturated monomer wherein the inhibitor is recycled back to the distillation column (see the Figure). Therefore, it would have been obvious to one having ordinary skill in the art at the time the invention was made to have modified the process of Arhancet by recycling the inhibitor back to the distillation column as taught by Higgins because the recycling step would cut down the cost of fresh inhibitor."

Higgins et al. disclose the inhibition of styrene polymerization during the distillation thereof by incorporating therein, in an amount sufficient to inhibit polymerization thereof, a *dinitrophenol* solution recovered from styrene still residues or tars resulting from the distillation of styrene in the presence of *dinitrophenol*.

Dinitrophenol is *not* a *nitroxyl-containing* compound and thus this patent provides no teaching of the difficulties encountered in using nitroxyl-containing compounds as inhibitors, nor does it suggest that problems involved in using nitroxyl-containing compounds as inhibitors can be overcome by recycling a stream *containing such inhibitors* at temperatures no higher than about 110°C *and* at pressures below 760 mm Hg, as required by the present claims. On the contrary, the patent teaches in column 4, at lines 38-41 that the *distillation column* was operated at an overhead pressure of 414 mm Hg which *resulted in a bottoms*

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temperature of approximately 131° C. This is precisely the kind of distillation temperature the present Applicants have taught is to be avoided.

Further, and even more important, Higgins et al. teach that by virtue of their recycling process, a solution of their recycled dinitrophenols is provided that is *more effective* than the commercial dinitrophenols ordinarily employed in retarding polymerization during the distillation of styrene. See column 1, lines 61-66 and column 4, Example 3. Thus, a person of ordinary skill in the art, aware that, as pointed out above, it is known in the industry that the recycling of streams utilizing nitroxyls as polymerization inhibitors in plants employing temperatures in excess of about 115°C causes *loss* of inhibitor efficiency, such that the tar recycle leads to a higher polymer content than would be expected or desirable, could hardly be expected to think of following the teaching of Higgins et al. to supplement the deficiencies of Arhancet. In other words, the behavior of dinitrophenols and nitroxyl compounds are so totally different that the skilled practitioner would have no motivation whatsoever to combine the teaching of Higgins et al. with the teaching of Arhancet to come up with the present invention.

The Examiner also stated during prosecution:

"Regarding claim 9, Arhancet does not specifically disclose that the distillation is a continuous operation. It would have been obvious to one having ordinary skill in the art at the time the invention was made to have modified the process of Arhancet by operating the process continuously because Higgins teaches that similar results would be expected when the distillation is operated in either continuous or batch mode."

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It is not to be denied that the use of batch and continuous operations are well-known in the art. However, the use of a continuous operation wherein the continuity includes the recycling of a nitroxyl inhibitor presents special problems with regard to effectiveness that one does not encounter with other inhibitors, such as dinitrophenols. Neither Arhancet nor Higgins et al., either alone or in combination, provides any teaching as to how these problems can be overcome.

Finally, the Examiner also stated:

"Regarding claim 18, Arhancet does not specifically disclose that the inhibitor is a blend of two nitroxyls. However, each of the nitroxyl-containing inhibitors of Arhancet has an equivalent function. Therefore, it would have been obvious to one having ordinary skill in the art at the time the invention was made to have modified the process of Arhancet by using a blend of two nitroxyls because it would be expected that the mixture of the two nitroxyls would have similar results as a single nitroxyl inhibitor."

In the real world, where economics is a supreme consideration, scientists and engineers don't go around looking for ways to make commercial processes more complex in the absence of countervailing benefits that make the added cost of the more complex system acceptable. Here, Arhancet has taught the use of a single nitroxyl compound; there would be no motivation for the person in charge of a styrene purification process to simply throw in a second or third nitroxyl compound, unless he was aware of some good reason for doing so in the light of the increased cost it would entail. No such good reason is provided by Arhancet.

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Conclusion

The Examiner's cited references fail to disclose or make obvious the improvement to the process claimed by the Appellants. These rejections should be reversed.

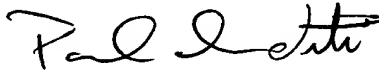
Favorable consideration of the application is respectfully requested.

9. Appendix

An Appendix is attached that contains a copy of the claims, as amended, that are involved in the appeal.

Respectfully submitted,

29 June 05
Date

For  Reg. No. 30,754
James L. Lewis
Registration No. 24,732

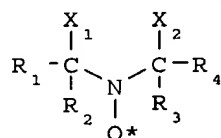
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APPENDIX

1. In a process for the production and purification of unsaturated monomers employing distillation means and a nitroxyl-containing polymerization inhibitor of said monomers, wherein a process stream containing the nitroxyl-containing inhibitor is removed downstream of the distillation means and returned to the process ahead of the distillation means, the improvement which comprises recycling said stream containing the nitroxyl-containing inhibitor into the distillation means, wherein the temperature in the distillation means is no higher than about 110° C and the pressure is less than 760 mm Hg.

2. The process of claim 1 wherein the nitroxyl-containing inhibitor is of the following structural formula:



wherein

R₁ and R₄ are independently selected from the group consisting of hydrogen, alkyl, and heteroatom-substituted alkyl;

R₂ and R₃ are independently selected from the group consisting of alkyl and heteroatom-substituted alkyl; and

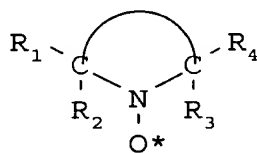
X₁ and X₂

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- (1) are independently selected from the group consisting of halogen, cyano, amido, $-S-C_6H_5$, carbonyl, alkenyl, alkyl of 1 to 15 carbon atoms, $COOR_7$, $-S-COR_7$, and $-OCOR_7$, wherein R_7 is alkyl or aryl, or
- (2) taken together, form a ring structure with the nitrogen.

9. The process of claim 1 wherein the distillation is a continuous operation.

17. The process of claim 2 wherein the nitroxyl-containing inhibitor is of the structure



wherein R_1 and R_4 are independently selected from the group consisting of hydrogen, alkyl, and heteroatom-substituted alkyl and R_2 and R_3 are independently selected from the group consisting of alkyl and heteroatom-substituted alkyl, and the



portion represents the atoms necessary to form a five-, six-, or seven-membered heterocyclic ring.

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18. The process of claim 2 wherein the inhibitor is a blend of two nitroxyls.
19. The process of claim 17 wherein the inhibitor contains one or more nitroxyls selected from the group consisting of:
- N,N-di-*tert*-butylnitroxide;
 - N,N-di-*tert*-amylnitroxide;
 - N-*tert*-butyl-2-methyl-1-phenyl-propylnitroxide;
 - N-*tert*-butyl-1-diethylphosphono-2,2-dimethylpropylnitroxide;
 - 2,2,6,6-tetramethyl-piperidinyloxy;
 - 4-amino-2,2,6,6-tetramethyl-piperidinyloxy;
 - 4-hydroxy-2,2,6,6-tetramethyl-piperidinyloxy;
 - 4-oxo-2,2,6,6-tetramethyl-piperidinyloxy;
 - 4-dimethylamino-2,2,6,6-tetramethyl-piperidinyloxy;
 - 4-ethanoyloxy-2,2,6,6-tetramethyl-piperidinyloxy;
 - 2,2,5,5-tetramethylpyrrolidinyloxy;
 - 3-amino-2,2,5,5-tetramethylpyrrolidinyloxy;
 - 2,2,4,4-tetramethyl-1-oxa-3-azacyclopentyl-3-oxy;
 - 2,2,4,4-tetramethyl-1-oxa-3-pyrrolinyl-1-oxy-3-carboxylic acid;
 - 2,2,3,3,5,5,6,6-octamethyl-1,4-diazacyclohexyl-1,4-dioxy;
 - 4-bromo-2,2,6,6-tetramethyl-piperidinyloxy;
 - 4-chloro-2,2,6,6-tetramethyl-piperidinyloxy;
 - 4-iodo-2,2,6,6-tetramethyl-piperidinyloxy;

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4-fluoro-2,2,6,6-tetramethyl-piperidinyloxy;
4-cyano-2,2,6,6-tetramethyl-piperidinyloxy;
4-carboxy-2,2,6,6-tetramethyl-piperidinyloxy;
4-carbomethoxy-2,2,6,6-tetramethyl-piperidinyloxy;
4-carbethoxy-2,2,6,6-tetramethyl-piperidinyloxy;
4-cyano-4-hydroxy-2,2,6,6-tetramethyl-piperidinyloxy;
4-methyl-2,2,6,6-tetramethyl-piperidinyloxy;
4-carbethoxy-4-hydroxy-2,2,6,6-tetramethyl-piperidinyloxy;
4-hydroxy-4-(1-hydroxypropyl)-2,2,6,6-tetramethyl-piperidinyloxy;
4-methyl-2,2,6,6-tetramethyl-1,2,5,6-tetrahydropyridine -1-oxyl;
4-carboxy-2,2,6,6-tetramethyl-1,2,5,6-tetrahydropyridine -1-oxyl;
4-carbomethoxy-2,2,6,6-tetramethyl-1,2,5,6-tetrahydropyridine -1-oxyl;
4-carbethoxy-2,2,6,6-tetramethyl-1,2,5,6-tetrahydropyridine -1-oxyl;
4-amino-2,2,6,6-tetramethyl-1,2,5,6-tetrahydropyridine -1-oxyl;
4-amido-2,2,6,6-tetramethyl-1,2,5,6-tetrahydropyridine -1-oxyl;
3,4-diketo-2,2,5,5-tetramethylpyrrolidinyloxy;
3-keto-4-oximino-2,2,5,5-tetramethylpyrrolidinyloxy;
3-keto-4-benzylidene-2,2,5,5-tetramethylpyrrolidinyloxy;
3-keto-4,4-dibromo-2,2,5,5-tetramethylpyrrolidinyloxy;
2,2,3,3,5,5-hexamethylpyrrolidinyloxy;
3-carboximido-2,2,5,5-tetramethylpyrrolidinyloxy;
3-oximino-2,2,5,5-tetramethylpyrrolidinyloxy;

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3-hydroxy-2,2,5,5-tetramethylpyrrolidinyloxy;
3-cyano-3-hydroxy-2,2,5,5-tetramethylpyrrolidinyloxy;
3-carbomethoxy-3-hydroxy-2,2,5,5-tetramethylpyrrolidinyloxy;
3-carbethoxy-3-hydroxy-2,2,5,5-tetramethylpyrrolidinyloxy;
2,2,5,5-tetramethyl-3-carboxamido-2,5-dihydropyrrole-1-oxyl;
2,2,5,5-tetramethyl-3-amino-2,5-dihydropyrrole-1-oxyl;
2,2,5,5-tetramethyl-3-carbethoxy-2,5-dihydropyrrole-1-oxyl;
2,2,5,5-tetramethyl-3-cyano-2,5-dihydropyrrole-1-oxyl;
bis(1-oxyl-2,2,6,6-tetramethylpiperidin-4-yl)succinate;
bis(1-oxyl-2,2,6,6-tetramethylpiperidin-4-yl)adipate;
bis(1-oxyl-2,2,6,6-tetramethylpiperidin-4-yl)sebacate;
bis(1-oxyl-2,2,6,6-tetramethylpiperidin-4-yl)n-butylmalonate;
bis(1-oxyl-2,2,6,6-tetramethylpiperidin-4-yl)phthalate;
bis(1-oxyl-2,2,6,6-tetramethylpiperidin-4-yl)isophthalate;
bis(1-oxyl-2,2,6,6-tetramethylpiperidin-4-yl)terephthalate;
bis(1-oxyl-2,2,6,6-tetramethylpiperidin-4-yl)hexahydroterephthalate;
N,N'-bis(1-oxyl-2,2,6,6-tetramethylpiperidin-4-yl)adipamide;
N-(1-oxyl-2,2,6,6-tetramethylpiperidin-4-yl)-caprolactam;
N-(1-oxyl-2,2,6,6-tetramethylpiperidin-4-yl)-dodecylsuccinimide;
2,4,6-tris-[N-butyl-N-(1-oxyl-2,2,6,6-tetramethylpiperidin-4-yl)]-s-triazine; and
4,4'-ethylenebis(1-oxyl-2,2,6,6-tetramethylpiperazin-3-one).

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20. The process of claim 1 wherein said monomers contain impurities from the monomer production and/or purification processes.
21. The process of claim 20 wherein the impurities include polymer formed during the production and/or purification processes.
22. The process of claim 21 wherein the polymer formed during the production and/or purification processes is soluble in the monomer.
23. The process of claim 21 wherein the polymer formed during the production and/or purification processes is insoluble in the monomer.
24. The process of claim 21 wherein the equipment in which the distillation process occurs contains polymer.
25. The process of claim 24 wherein the polymer was formed during the monomer's production and/or purification processes.
26. The process of claim 24 wherein the polymer is not dissolved in the monomer.

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Dinitrophenol is *not* a *nitroxyl-containing* compound and thus this patent provides no teaching of the difficulties encountered in using nitroxyl-containing compounds as inhibitors, nor does it suggest that problems involved in using nitroxyl-containing compounds as inhibitors can be overcome by recycling a stream *containing such inhibitors* at temperatures no higher than about 110°C *and* at pressures below 760 mm Hg, as required by the present claims. On the contrary, the patent teaches in column 4, at lines 38-41 that the *distillation column* was operated at an overhead pressure of 414 mm Hg which *resulted in a bottoms*

temperature of approximately 131° C. This is precisely the kind of distillation temperature the present Applicants have taught is to be avoided.

Further, and even more important, Higgins et al. teach that by virtue of their recycling process, a solution of their recycled dinitrophenols is provided that is *more effective* than the commercial dinitrophenols ordinarily employed in retarding polymerization during the distillation of styrene. See column 1, lines 61-66 and column 4, Example 3. Thus, a person of ordinary skill in the art, aware that, as pointed out above, it is known in the industry that the recycling of streams utilizing nitroxyls as polymerization inhibitors in plants employing temperatures in excess of about 115°C causes *loss* of inhibitor efficiency, such that the tar recycle leads to a higher polymer content than would be expected or desirable, could hardly be expected to think of following the teaching of Higgins et al. to supplement the deficiencies of Arhancet. In other words, the behavior of dinitrophenols and nitroxyl compounds are so totally different that the skilled practitioner would have no motivation whatsoever to combine the teaching of Higgins et al. with the teaching of Arhancet to come up with the present invention.

The Examiner also stated during prosecution:

"Regarding claim 9, Arhancet does not specifically disclose that the distillation is a continuous operation. It would have been obvious to one having ordinary skill in the art at the time the invention was made to have modified the process of Arhancet by operating the process continuously because Higgins teaches that similar results would be expected when the distillation is operated in either continuous or batch mode."

It is not to be denied that the use of batch and continuous operations are well-known in the art. However, the use of a continuous operation wherein the continuity includes the recycling of a nitroxyl inhibitor presents special problems with regard to effectiveness that one does not encounter with other inhibitors, such as dinitrophenols. Neither Arhancet nor Higgins et al., either alone or in combination, provides any teaching as to how these problems can be overcome.

Finally, the Examiner also stated:

"Regarding claim 18, Arhancet does not specifically disclose that the inhibitor is a blend of two nitroxyls. However, each of the nitroxyl-containing inhibitors of Arhancet has an equivalent function. Therefore, it would have been obvious to one having ordinary skill in the art at the time the invention was made to have modified the process of Arhancet by using a blend of two nitroxyls because it would be expected that the mixture of the two nitroxyls would have similar results as a single nitroxyl inhibitor."

In the real world, where economics is a supreme consideration, scientists and engineers don't go around looking for ways to make commercial processes more complex in the absence of countervailing benefits that make the added cost of the more complex system acceptable. Here, Arhancet has taught the use of a single nitroxyl compound; there would be no motivation for the person in charge of a styrene purification process to simply throw in a second or third nitroxyl compound, unless he was aware of some good reason for doing so in the light of the increased cost it would entail. No such good reason is provided by Arhancet.

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Conclusion

The Examiner's cited references fail to disclose or make obvious the improvement to the process claimed by the Appellants. These rejections should be reversed.

Favorable consideration of the application is respectfully requested.

9. Appendix

An Appendix is attached that contains a copy of the claims, as amended, that are involved in the appeal.

Respectfully submitted,

29 June 05
Date

Paul I. Latta Reg. No. 30,754
for James L. Lewis
Registration No. 24,732

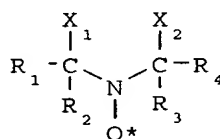
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APPENDIX

1. In a process for the production and purification of unsaturated monomers employing distillation means and a nitroxyl-containing polymerization inhibitor of said monomers, wherein a process stream containing the nitroxyl-containing inhibitor is removed downstream of the distillation means and returned to the process ahead of the distillation means, the improvement which comprises recycling said stream containing the nitroxyl-containing inhibitor into the distillation means, wherein the temperature in the distillation means is no higher than about 110° C and the pressure is less than 760 mm Hg.

2. The process of claim 1 wherein the nitroxyl-containing inhibitor is of the following structural formula:



wherein

R₁ and R₄ are independently selected from the group consisting of hydrogen, alkyl, and heteroatom-substituted alkyl;

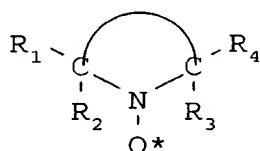
R₂ and R₃ are independently selected from the group consisting of alkyl and heteroatom-substituted alkyl; and

X₁ and X₂

- (1) are independently selected from the group consisting of halogen, cyano, amido, $-S-C_6H_5$, carbonyl, alkenyl, alkyl of 1 to 15 carbon atoms, $COOR_7$, $-S-COR_7$, and $-OCOR_7$, wherein R_7 is alkyl or aryl, or
- (2) taken together, form a ring structure with the nitrogen.

9. The process of claim 1 wherein the distillation is a continuous operation.

17. The process of claim 2 wherein the nitroxyl-containing inhibitor is of the structure



wherein R_1 and R_4 are independently selected from the group consisting of hydrogen, alkyl, and heteroatom-substituted alkyl and R_2 and R_3 are independently selected from the group consisting of alkyl and heteroatom-substituted alkyl, and the



portion represents the atoms necessary to form a five-, six-, or seven-membered heterocyclic ring.

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18. The process of claim 2 wherein the inhibitor is a blend of two nitroxyls.
19. The process of claim 17 wherein the inhibitor contains one or more nitroxyls selected from the group consisting of:
- N,N-di-*tert*-butylnitroxide;
- N,N-di-*tert*-amylnitroxide;
- N-*tert*-butyl-2-methyl-1-phenyl-propylnitroxide;
- N-*tert*-butyl-1-diethylphosphono-2,2-dimethylpropylnitroxide;
- 2,2,6,6-tetramethyl-piperidinyloxy;
- 4-amino-2,2,6,6-tetramethyl-piperidinyloxy;
- 4-hydroxy-2,2,6,6-tetramethyl-piperidinyloxy;
- 4-oxo-2,2,6,6-tetramethyl-piperidinyloxy;
- 4-dimethylamino-2,2,6,6-tetramethyl-piperidinyloxy;
- 4-ethanoyloxy-2,2,6,6-tetramethyl-piperidinyloxy;
- 2,2,5,5-tetramethylpyrrolidinyloxy;
- 3-amino-2,2,5,5-tetramethylpyrrolidinyloxy;
- 2,2,4,4-tetramethyl-1-oxa-3-azacyclopentyl-3-oxy;
- 2,2,4,4-tetramethyl-1-oxa-3-pyrrolinyl-1-oxy-3-carboxylic acid;
- 2,2,3,3,5,5,6,6-octamethyl-1,4-diazacyclohexyl-1,4-dioxy;
- 4-bromo-2,2,6,6-tetramethyl-piperidinyloxy;
- 4-chloro-2,2,6,6-tetramethyl-piperidinyloxy;
- 4-iodo-2,2,6,6-tetramethyl-piperidinyloxy;

4-fluoro-2,2,6,6-tetramethyl-piperidinyloxy;

4-cyano-2,2,6,6-tetramethyl-piperidinyloxy;

4-carboxy-2,2,6,6-tetramethyl-piperidinyloxy;

4-carbomethoxy-2,2,6,6-tetramethyl-piperidinyloxy;

4-carbethoxy-2,2,6,6-tetramethyl-piperidinyloxy;

4-cyano-4-hydroxy-2,2,6,6-tetramethyl-piperidinyloxy;

4-methyl-2,2,6,6-tetramethyl-piperidinyloxy;

4-carbethoxy-4-hydroxy-2,2,6,6-tetramethyl-piperidinyloxy;

4-hydroxy-4-(1-hydroxypropyl)-2,2,6,6-tetramethyl-piperidinyloxy;

4-methyl-2,2,6,6-tetramethyl-1,2,5,6-tetrahydropyridine -1-oxyl;

4-carboxy-2,2,6,6-tetramethyl-1,2,5,6-tetrahydropyridine -1-oxyl;

4-carbomethoxy-2,2,6,6-tetramethyl-1,2,5,6-tetrahydropyridine -1-oxyl;

4-carbethoxy-2,2,6,6-tetramethyl-1,2,5,6-tetrahydropyridine -1-oxyl;

4-amino-2,2,6,6-tetramethyl-1,2,5,6-tetrahydropyridine -1-oxyl;

4-amido-2,2,6,6-tetramethyl-1,2,5,6-tetrahydropyridine -1-oxyl;

3,4-diketo-2,2,5,5-tetramethylpyrrolidinyloxy;

3-keto-4-oximino-2,2,5,5-tetramethylpyrrolidinyloxy;

3-keto-4-benzylidene-2,2,5,5-tetramethylpyrrolidinyloxy;

3-keto-4,4-dibromo-2,2,5,5-tetramethylpyrrolidinyloxy;

2,2,3,3,5,5-hexamethylpyrrolidinyloxy;

3-carboximido-2,2,5,5-tetramethylpyrrolidinyloxy;

3-oximino-2,2,5,5-tetramethylpyrrolidinyloxy;

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3-hydroxy-2,2,5,5-tetramethylpyrrolidinyloxy;
3-cyano-3-hydroxy-2,2,5,5-tetramethylpyrrolidinyloxy;
3-carbomethoxy-3-hydroxy-2,2,5,5-tetramethylpyrrolidinyloxy;
3-carbethoxy-3-hydroxy-2,2,5,5-tetramethylpyrrolidinyloxy;
2,2,5,5-tetramethyl-3-carboxamido-2,5-dihydropyrrole-1-oxyl;
2,2,5,5-tetramethyl-3-amino-2,5-dihydropyrrole-1-oxyl;
2,2,5,5-tetramethyl-3-carbethoxy-2,5-dihydropyrrole-1-oxyl;
2,2,5,5-tetramethyl-3-cyano-2,5-dihydropyrrole-1-oxyl;
bis(1-oxyl-2,2,6,6-tetramethylpiperidin-4-yl)succinate;
bis(1-oxyl-2,2,6,6-tetramethylpiperidin-4-yl)adipate;
bis(1-oxyl-2,2,6,6-tetramethylpiperidin-4-yl)sebacate;
bis(1-oxyl-2,2,6,6-tetramethylpiperidin-4-yl)n-butylmalonate;
bis(1-oxyl-2,2,6,6-tetramethylpiperidin-4-yl)phthalate;
bis(1-oxyl-2,2,6,6-tetramethylpiperidin-4-yl)isophthalate;
bis(1-oxyl-2,2,6,6-tetramethylpiperidin-4-yl)terephthalate;
bis(1-oxyl-2,2,6,6-tetramethylpiperidin-4-yl)hexahydroterephthalate;
N,N'-bis(1-oxyl-2,2,6,6-tetramethylpiperidin-4-yl)adipamide;
N-(1-oxyl-2,2,6,6-tetramethylpiperidin-4-yl)-caprolactam;
N-(1-oxyl-2,2,6,6-tetramethylpiperidin-4-yl)-dodecylsuccinimide;
2,4,6-tris-[N-butyl-N-(1-oxyl-2,2,6,6-tetramethylpiperidin-4-yl)]-s-triazine; and
4,4'-ethylenebis(1-oxyl-2,2,6,6-tetramethylpiperazin-3-one).

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20. The process of claim 1 wherein said monomers contain impurities from the monomer production and/or purification processes.
21. The process of claim 20 wherein the impurities include polymer formed during the production and/or purification processes.
22. The process of claim 21 wherein the polymer formed during the production and/or purification processes is soluble in the monomer.
23. The process of claim 21 wherein the polymer formed during the production and/or purification processes is insoluble in the monomer.
24. The process of claim 21 wherein the equipment in which the distillation process occurs contains polymer.
25. The process of claim 24 wherein the polymer was formed during the monomer's production and/or purification processes.
26. The process of claim 24 wherein the polymer is not dissolved in the monomer.

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COPY

**PATENT
IN THE UNITED STATES PATENT AND TRADEMARK OFFICE
BEFORE THE BOARD OF PATENT APPEALS AND INTERFERENCES**

Appl. No. : 09/910,968
Applicant : Brigitte BENAGE et al.
Filed : July 23, 2001
Title : RECYCLE OF NITROXYL-CONTAINING STREAMS AT LOW
TEMPERATURE
TC/A.U. : 1764
Examiner: : Nguyen, T.M.
Docket No. : 0036-PA (UNI057US)

APPELLANT'S BRIEF

Sir:

The above-identified Appellants submit this Appellant's Brief in triplicate pursuant to 37 C.F.R. § 1.192. The Notice of Appeal was filed on May 13, 2005.

Please charge the official fee of \$500.00 for filing a brief in support of an appeal to our Deposit Account Number 23-2656. A duplicate copy of this page is enclosed.

The Appellant relies upon the following authorities and arguments to maintain the appeal.

1. Real Party In Interest

The real party in interest for this matter is the Appellant's assignee. The assignee and real party in interest is Crompton Corporation; Benson Road; Middlebury, CT 06749.

2. Related Appeals and Interferences

There are no other appeals or interferences that will directly affect or be directly affected by or have a bearing on the Board's decision in the pending appeal.

3. Status of Claims

A Provisional Application, Application No. 60/222,595 was filed on August 2, 2000. The Non-Provisional application based thereon was filed July 23, 2001.

Claims 1 through 19 were originally filed with the application.

By amendment filed December 30, 2002, Appellant canceled claims 1-3, 17 and 18, amended claims 4-16 and 19, and added new claim 31.

By amendment under 37 C.F.R. § 1.116 filed April 28, 2003, Appellant attempted to amend claims 4, 16, 19, and 31. In an Advisory Action dated June 2, 2003, the Examiner indicated that the proposed amendments would not be entered on the ground that they raised new issues that would require further consideration and/or search.

On June 18, 2003, Appellant filed a Request for Continued Examination.

In an Office Action dated July 2, 2003, the amendments presented on April 28, 2003 were rejected.

Appellant amended claims 16 and 31 in an Amendment filed September 15, 2003.

In an Office Action dated November 18, 2003, claims 4-16 and 19-31 were finally rejected.

4. Status of Amendments

All amendments filed in the application have been entered.

5. Summary of Invention

The present invention is directed to an improvement in a process for the production and purification of unsaturated monomers employing distillation means and a nitroxyl-containing polymerization inhibitor of said monomers, wherein a process stream containing the nitroxyl-containing inhibitor is removed downstream of the distillation means and returned to the process ahead of the distillation means, wherein the improvement comprises recycling said stream containing the nitroxyl-containing inhibitor into the distillation means, wherein the temperature in the distillation means is no higher than about 110° C and the pressure is less than 760 mm Hg.

See pending claim 1, the specification at page 14, lines 1-5, and claim 8 of the application as originally filed.

6. Issue

Are claims 1, 2, 9, and 17-26 unpatentable over Arhancet (U.S. Patent No. 5,907,071) in view of Higgins et al. (U.S. Patent No. 4,033,829) under 35 U.S.C. 103(a)?

7. **Grouping of Claims**

All claims 1, 2, 9, and 17-26 are of a single group that is directed to an improvement in a process for the production and purification of unsaturated monomers employing distillation means and a nitroxyl-containing polymerization inhibitor of said monomers, wherein a process stream containing the nitroxyl-containing inhibitor is removed downstream of the distillation means and returned to the process ahead of the distillation means, wherein the improvement comprises recycling said stream containing the nitroxyl-containing inhibitor into the distillation means, wherein the temperature in the distillation means is no higher than about 110° C and the pressure is less than 760 mm Hg.

All the claims of the rejected group of claims stand or fall together.

8. **Argument**

Issue: Are claims 1, 2, 9, and 17-26 unpatentable over Arhancet (U.S. Patent No. 5,907,071) in view of Higgins et al. (U.S. Patent No. 4,033,829) under 35 U.S.C. 103(a)?

Arhancet discloses the inhibition of the polymerization of vinyl aromatic monomers such as styrene by the addition of a composition of a stable hindered nitroxyl radical and an oxime compound.

The Examiner has stated during prosecution:

"Arhancet discloses a method for inhibiting premature polymerization of vinyl aromatic monomers. The monomers are distilled in the presence of nitroxyl inhibitors (2,2,6,6-tetramethyl-1-piperidinoxy) at a temperature of 110° C and under

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reduced vacuum. The formula of the inhibitor of Arhancet is the same as the claimed formula. (See col. 1, lines 16-17; col. 2, line 10 through col. 3, line 7; claims 6 and 8). ...

Regarding claim 1, Arhancet does not disclose a step of recycling the inhibitor to the distillation column."

This last sentence is precisely the point. Arhancet teaches only using fresh nitroxyl compounds (i.e., not recycled) in combination with an oxime compound. As pointed out on page 13 of the present application, at lines 20-23,

"It is known in the industry that the recycling of streams utilizing nitroxyls as polymerization inhibitors in plants employing temperatures in excess of about 115°C causes loss of inhibitor efficiency, such that the tar recycle leads to a higher polymer content than would be expected or desirable."

The present invention is directed to the discovery of a means to substantially overcome this problem of the decreased efficiency of recycled *nitroxyl* inhibitors.

In an attempt to overcome this deficiency of Arhancet as a reference, the Examiner has cited the Higgins et al. reference.

According to the Examiner:

"Higgins discloses a process for production/purification of an unsaturated monomer feedstock by contacting the feedstock with inhibitors in a distillation/separation zone to produce a product stream containing the inhibitor and the unsaturated monomer which is then recycled back to the distillation/separation zone. Higgins also discloses that the process is operated on either a continuous or batch basis at an overhead pressure of the distillation

column of 414 mm Hg and the product stream contains by-product impurities such as polymers. (See abstract; col. 1, lines 46-64; col. 3, line 11 through col. 6, line 66)

Regarding claim 1, Arhancet does not disclose a step of recycling the inhibitor to the distillation column. However, Higgins discloses a process for production/purification of an unsaturated monomer wherein the inhibitor is recycled back to the distillation column (see the Figure). Therefore, it would have been obvious to one having ordinary skill in the art at the time the invention was made to have modified the process of Arhancet by recycling the inhibitor back to the distillation column as taught by Higgins because the recycling step would cut down the cost of fresh inhibitor."

Higgins et al. disclose the inhibition of styrene polymerization during the distillation thereof by incorporating therein, in an amount sufficient to inhibit polymerization thereof, a *dinitrophenol* solution recovered from styrene still residues or tars resulting from the distillation of styrene in the presence of *dinitrophenol*.

Dinitrophenol is *not* a *nitroxyl-containing* compound and thus this patent provides no teaching of the difficulties encountered in using nitroxyl-containing compounds as inhibitors, nor does it suggest that problems involved in using nitroxyl-containing compounds as inhibitors can be overcome by recycling a stream *containing such inhibitors* at temperatures no higher than about 110°C *and* at pressures below 760 mm Hg, as required by the present claims. On the contrary, the patent teaches in column 4, at lines 38-41 that the *distillation column* was operated at an overhead pressure of 414 mm Hg which *resulted in a bottoms*

temperature of approximately 131° C. This is precisely the kind of distillation temperature the present Applicants have taught is to be avoided.

Further, and even more important, Higgins et al. teach that by virtue of their recycling process, a solution of their recycled dinitrophenols is provided that is *more effective* than the commercial dinitrophenols ordinarily employed in retarding polymerization during the distillation of styrene. See column 1, lines 61-66 and column 4, Example 3. Thus, a person of ordinary skill in the art, aware that, as pointed out above, it is known in the industry that the recycling of streams utilizing nitroxyls as polymerization inhibitors in plants employing temperatures in excess of about 115°C causes *loss* of inhibitor efficiency, such that the tar recycle leads to a higher polymer content than would be expected or desirable, could hardly be expected to think of following the teaching of Higgins et al. to supplement the deficiencies of Arhancet. In other words, the behavior of dinitrophenols and nitroxyl compounds are so totally different that the skilled practitioner would have no motivation whatsoever to combine the teaching of Higgins et al. with the teaching of Arhancet to come up with the present invention.

The Examiner also stated during prosecution:

"Regarding claim 9, Arhancet does not specifically disclose that the distillation is a continuous operation. It would have been obvious to one having ordinary skill in the art at the time the invention was made to have modified the process of Arhancet by operating the process continuously because Higgins teaches that similar results would be expected when the distillation is operated in either continuous or batch mode."

It is not to be denied that the use of batch and continuous operations are well-known in the art. However, the use of a continuous operation wherein the continuity includes the recycling of a nitroxyl inhibitor presents special problems with regard to effectiveness that one does not encounter with other inhibitors, such as dinitrophenols. Neither Arhancet nor Higgins et al., either alone or in combination, provides any teaching as to how these problems can be overcome.

Finally, the Examiner also stated:

"Regarding claim 18, Arhancet does not specifically disclose that the inhibitor is a blend of two nitroxyls. However, each of the nitroxyl-containing inhibitors of Arhancet has an equivalent function. Therefore, it would have been obvious to one having ordinary skill in the art at the time the invention was made to have modified the process of Arhancet by using a blend of two nitroxyls because it would be expected that the mixture of the two nitroxyls would have similar results as a single nitroxyl inhibitor."

In the real world, where economics is a supreme consideration, scientists and engineers don't go around looking for ways to make commercial processes more complex in the absence of countervailing benefits that make the added cost of the more complex system acceptable. Here, Arhancet has taught the use of a single nitroxyl compound; there would be no motivation for the person in charge of a styrene purification process to simply throw in a second or third nitroxyl compound, unless he was aware of some good reason for doing so in the light of the increased cost it would entail. No such good reason is provided by Arhancet.

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An Appendix is attached that contains a copy of the claims, as amended, that are involved in the appeal.

Respectfully submitted,

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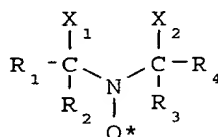
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APPENDIX

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2. The process of claim 1 wherein the nitroxyl-containing inhibitor is of the following structural formula:



wherein

R₁ and R₄ are independently selected from the group consisting of hydrogen, alkyl, and heteroatom-substituted alkyl;

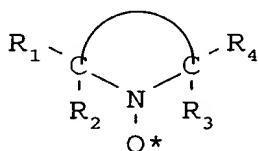
R₂ and R₃ are independently selected from the group consisting of alkyl and heteroatom-substituted alkyl; and

X₁ and X₂

- (1) are independently selected from the group consisting of halogen, cyano, amido, $-S-C_6H_5$, carbonyl, alkenyl, alkyl of 1 to 15 carbon atoms, $COOR_7$, $-S-COR_7$, and $-OCOR_7$, wherein R_7 is alkyl or aryl, or
- (2) taken together, form a ring structure with the nitrogen.

9. The process of claim 1 wherein the distillation is a continuous operation.

17. The process of claim 2 wherein the nitroxyl-containing inhibitor is of the structure



wherein R_1 and R_4 are independently selected from the group consisting of hydrogen, alkyl, and heteroatom-substituted alkyl and R_2 and R_3 are independently selected from the group consisting of alkyl and heteroatom-substituted alkyl, and the



portion represents the atoms necessary to form a five-, six-, or seven-membered heterocyclic ring.

18. The process of claim 2 wherein the inhibitor is a blend of two nitroxyls.

19. The process of claim 17 wherein the inhibitor contains one or more nitroxyls selected from the group consisting of:

N,N-di-*tert*-butylnitroxide;

N,N-di-*tert*-amylnitroxide;

N-*tert*-butyl-2-methyl-1-phenyl-propylnitroxide;

N-*tert*-butyl-1-diethylphosphono-2,2-dimethylpropylnitroxide;

2,2,6,6-tetramethyl-piperidinyloxy;

4-amino-2,2,6,6-tetramethyl-piperidinyloxy;

4-hydroxy-2,2,6,6-tetramethyl-piperidinyloxy;

4-oxo-2,2,6,6-tetramethyl-piperidinyloxy;

4-dimethylamino-2,2,6,6-tetramethyl-piperidinyloxy;

4-ethanoyloxy-2,2,6,6-tetramethyl-piperidinyloxy;

2,2,5,5-tetramethylpyrrolidinyloxy;

3-amino-2,2,5,5-tetramethylpyrrolidinyloxy;

2,2,4,4-tetramethyl-1-oxa-3-azacyclopentyl-3-oxy;

2,2,4,4-tetramethyl-1-oxa-3-pyrrolinyl-1-oxy-3-carboxylic acid;

2,2,3,3,5,5,6,6-octamethyl-1,4-diazacyclohexyl-1,4-dioxy;

4-bromo-2,2,6,6-tetramethyl-piperidinyloxy;

4-chloro-2,2,6,6-tetramethyl-piperidinyloxy;

4-iodo-2,2,6,6-tetramethyl-piperidinyloxy;

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4-fluoro-2,2,6,6-tetramethyl-piperidinyloxy;

4-cyano-2,2,6,6-tetramethyl-piperidinyloxy;

4-carboxy-2,2,6,6-tetramethyl-piperidinyloxy;

4-carbomethoxy-2,2,6,6-tetramethyl-piperidinyloxy;

4-carbethoxy-2,2,6,6-tetramethyl-piperidinyloxy;

4-cyano-4-hydroxy-2,2,6,6-tetramethyl-piperidinyloxy;

4-methyl-2,2,6,6-tetramethyl-piperidinyloxy;

4-carbethoxy-4-hydroxy-2,2,6,6-tetramethyl-piperidinyloxy;

4-hydroxy-4-(1-hydroxypropyl)-2,2,6,6-tetramethyl-piperidinyloxy;

4-methyl-2,2,6,6-tetramethyl-1,2,5,6-tetrahydropyridine -1-oxyl;

4-carboxy-2,2,6,6-tetramethyl-1,2,5,6-tetrahydropyridine -1-oxyl;

4-carbomethoxy-2,2,6,6-tetramethyl-1,2,5,6-tetrahydropyridine -1-oxyl;

4-carbethoxy-2,2,6,6-tetramethyl-1,2,5,6-tetrahydropyridine -1-oxyl;

4-amino-2,2,6,6-tetramethyl-1,2,5,6-tetrahydropyridine -1-oxyl;

4-amido-2,2,6,6-tetramethyl-1,2,5,6-tetrahydropyridine -1-oxyl;

3,4-diketo-2,2,5,5-tetramethylpyrrolidinyloxy;

3-keto-4-oximino-2,2,5,5-tetramethylpyrrolidinyloxy;

3-keto-4-benzylidene-2,2,5,5-tetramethylpyrrolidinyloxy;

3-keto-4,4-dibromo-2,2,5,5-tetramethylpyrrolidinyloxy;

2,2,3,3,5,5-hexamethylpyrrolidinyloxy;

3-carboximido-2,2,5,5-tetramethylpyrrolidinyloxy;

3-oximino-2,2,5,5-tetramethylpyrrolidinyloxy;

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3-hydroxy-2,2,5,5-tetramethylpyrrolidinyloxy;
3-cyano-3-hydroxy-2,2,5,5-tetramethylpyrrolidinyloxy;
3-carbomethoxy-3-hydroxy-2,2,5,5-tetramethylpyrrolidinyloxy;
3-carbethoxy-3-hydroxy-2,2,5,5-tetramethylpyrrolidinyloxy;
2,2,5,5-tetramethyl-3-carboxamido-2,5-dihydropyrrole-1-oxyl;
2,2,5,5-tetramethyl-3-amino-2,5-dihydropyrrole-1-oxyl;
2,2,5,5-tetramethyl-3-carbethoxy-2,5-dihydropyrrole-1-oxyl;
2,2,5,5-tetramethyl-3-cyano-2,5-dihydropyrrole-1-oxyl;
bis(1-oxyl-2,2,6,6-tetramethylpiperidin-4-yl)succinate;
bis(1-oxyl-2,2,6,6-tetramethylpiperidin-4-yl)adipate;
bis(1-oxyl-2,2,6,6-tetramethylpiperidin-4-yl)sebacate;
bis(1-oxyl-2,2,6,6-tetramethylpiperidin-4-yl)n-butylmalonate;
bis(1-oxyl-2,2,6,6-tetramethylpiperidin-4-yl)phthalate;
bis(1-oxyl-2,2,6,6-tetramethylpiperidin-4-yl)isophthalate;
bis(1-oxyl-2,2,6,6-tetramethylpiperidin-4-yl)terephthalate;
bis(1-oxyl-2,2,6,6-tetramethylpiperidin-4-yl)hexahydroterephthalate;
N,N'-bis(1-oxyl-2,2,6,6-tetramethylpiperidin-4-yl)adipamide;
N-(1-oxyl-2,2,6,6-tetramethylpiperidin-4-yl)-caprolactam;
N-(1-oxyl-2,2,6,6-tetramethylpiperidin-4-yl)-dodecylsuccinimide;
2,4,6-tris-[N-butyl-N-(1-oxyl-2,2,6,6-tetramethylpiperidin-4-yl)]-s-triazine; and
4,4'-ethylenebis(1-oxyl-2,2,6,6-tetramethylpiperazin-3-one).

20. The process of claim 1 wherein said monomers contain impurities from the monomer production and/or purification processes.

21. The process of claim 20 wherein the impurities include polymer formed during the production and/or purification processes.

22. The process of claim 21 wherein the polymer formed during the production and/or purification processes is soluble in the monomer.

23. The process of claim 21 wherein the polymer formed during the production and/or purification processes is insoluble in the monomer.

24. The process of claim 21 wherein the equipment in which the distillation process occurs contains polymer.

25. The process of claim 24 wherein the polymer was formed during the monomer's production and/or purification processes.

26. The process of claim 24 wherein the polymer is not dissolved in the monomer.